



PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
PATENT EXAMINING OPERATION

Applicant(s): James Edward MacDougall et al.

Serial No: 10/046,434

Group Art Unit: 1775

Filed: January 14, 2002

Examiner: Stephen J. Stein

Att. Docket No.: 05977PD USA

Confirmation No.: 3447

For: MESOPOROUS FILMS HAVING REDUCED DIELECTRIC CONSTANTS

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TC 1700

**DECLARATION UNDER 37 C.F.R. § 1.132**

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

CERTIFICATE OF MAILING	
I CERTIFY THAT THIS PAPER (ALONG WITH ANY PAPER REFERRED TO AS BEING ATTACHED OR ENCLOSED) IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE WITH SUFFICIENT POSTAGE AS FIRST CLASS MAIL IN AN ENVELOPE ADDRESSED TO: COMMISSIONER FOR PATENTS, P.O. BOX 1450, ALEXANDRIA, VA 22313-1450	
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Geoffrey L Chase	
Signature of person mailing paper	

Sir:

I, James E. MacDougall, Ph.D., a citizen of the United States of America, hereby declare and state:

1. The resume attached as Exhibit A accurately reflects my professional credentials.
2. I am a joint inventor named in the above-identified application.
3. I am an employee of Air Products and Chemicals Inc. (APCI), the assignee of the present application.
4. I understand from my review of the October 23, 2003 Final Rejection that claims of the present application were rejected as being anticipated by U.S. Patent No. 5,858,457 to Brinker et al. and as being anticipated or rendered obvious by U.S. Patent No. 5,645,891 to Liu et al.
5. I further understand from my review of the Final Rejection that the rejections can be obviated by "evidence showing that the prior art products do not necessarily possess the characteristics of the claimed products."
6. Independent claim 18 of this application is directed to a ceramic film produced by a specified process, wherein the ceramic film has a dielectric constant below 2.3, and a metal content of less than 500 ppm. Independent claim 27 is directed to a ceramic film having a dielectric constant below 2.3, and a metal content of less than 500 ppm. The tests described below, which were performed by an outside analytical lab, show that these claimed characteristics are not inherent features of the films of Brinker et al. or the films of Liu et al.

7. The tests employed VPD-ICP-MS (Vapor Phase Decomposition – Inductively Coupled Plasma – Mass Spectrometry) to analyze films prepared with and without purifying the surfactant. VPD-ICP-MS is a standard test in the industry for analyzing residual impurities on wafers. The data are representative of any material containing non-volatile impurities since this is a test to determine impurities for on-wafer applications. The surfactant will decompose and the volatile products will be removed in the vapor phase, but non-volatile metallic impurities from the surfactant will remain on the wafer as demonstrated by these tests.

8. The instant application at Table 14 lists some information about metal contents in the starting unpurified surfactants. We find that, for example, the starting potassium concentration in BASF L121 is about 483.1 ppm. This concentration of metal in the surfactant used to produce a film of the claimed invention having a thickness of about 6600 Å would yield a potassium concentration of  $51000 \times 10^{10}$  atoms/cm<sup>2</sup>. The conversion of this value to the ppm units specified in the claims at issue is as follows:

$$\begin{aligned} & (51000 \times 10^{10} \text{ K atoms/cm}^2 \text{ film}) \times (1/6600 \text{ Å film}) \times (1 \text{ mole K}/6.02 \times 10^{23} \text{ atoms K}) \times \\ & (39.1 \text{ g K/mole}) \times (\text{cm}^3 \text{ film}/0.9 \text{ g film} \times 10^8 \text{ Å/cm}) = 0.000558 \text{ g K/g film} \\ & (0.000558 \text{ g K/g film}) \times (1 \times 10^6) = 558 \text{ ppm.} \end{aligned}$$

9. Brinker et al. and Liu et al. both teach the use of cetyltrimethylammonium salts (chloride or bromide) as surfactants. ICP-MS (Inductively Coupled Plasma – Mass Spectrometry) analyses were conducted to determine the amount of alkali metal present in typical surfactants, including cetyltrimethylammonium chloride (CTAC). The amount of Na present in CTAC is 0.115 wt%. Since 1 wt% equals 10000 ppm, the amount of Na present is 1150 ppm for this surfactant. This sodium concentration is of the same order of magnitude as the potassium concentration determined for the L121 surfactant, discussed above. As we have demonstrated, if the starting surfactant contains large amounts of alkali metal, the metal content will carry forward to the film, resulting in a film containing large amounts of alkali metal.

10. A person having ordinary skill in the art would therefore anticipate that the films prepared by Brinker et al. or Liu et al. using a cetyltrimethylammonium salt surfactant would result in films with about 1000 ppm or more alkali metal present, based on the relationship between contaminants in surfactants and contaminants in films produced from the surfactants.

Since 483 ppm metal in the surfactant yields about 558 ppm metal in the film (as discussed in paragraph 8 above), then 1150 ppm metal in the surfactant would be expected to yield about 1328 ppm metal in a film of a similar thickness produced using a similar amount of surfactant.

11. Thus, neither Brinker et al. nor Liu et al. explicitly or inherently discloses ceramic film having a dielectric constant below 2.3, and a metal content of less than 500 ppm, as recited in independent claims 18 and 27 of the instant application.

12. Although Liu et al. teaches the desirability of removing halide from a film (not very effectively though – see column 4, lines 53-60 and column 5, lines 28-44), it does not disclose or suggest removing alkali metal from a film, and would not have motivated one of ordinary skill in the art to remove alkali metal from a film or use a surfactant with a low enough amount of alkali metal to avoid creating a film having a metal content less than 500 ppm. To the contrary, Liu et al. teaches away from such low metal content films in Examples 1-4 and 8, wherein alkali metal compounds are added as reagents (sodium aluminate) to produce the ceramic films.

13. Thus, one of ordinary skill in the art would not have found it obvious from the teachings of Liu et al. to produce a ceramic film having a dielectric constant below 2.3, and a metal content of less than 500 ppm, as recited in independent claims 18 and 27 of the instant application.

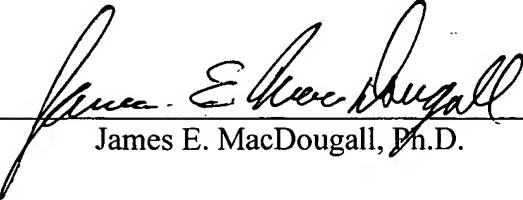
14. Regarding the issue of Bragg diffraction, the Final Rejection states that although Liu et al. is silent regarding the Bragg diffraction of its films, it would have been obvious to vary the components of the film forming composition to provide a film having the desired Bragg diffraction characteristics. Liu et al. discloses that its films are prepared by substantially using the method of U.S. Patent No. 5,098,684 to Kresge et al., but modified to make a film. Kresge et al. presents definitive evidence of preparing ordered materials that do show Bragg diffraction peaks. This is a desired result of the process, and one of ordinary skill in the art would have lacked reasonable motivation to alter the process to prepare more disordered materials (i.e., materials lacking Bragg diffraction), particularly in view of the understanding in the art at the time of the present invention that having order improves mechanical properties of films.

Application No. 10/046,434

Rule 132 Declaration of James E. MacDougall, Ph.D.

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine and/or imprisonment under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Date: 1/22/04

  
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**Goal:** To obtain a challenging industrial research position involving synthesis and modification of materials.

**Education**

Ph.D. in Inorganic Chemistry, The University of California - Santa Barbara, 5/90 Advisor: Professor Galen D. Stucky

B. S. in Chemistry, The Pennsylvania State University, 12/85  
Senior Research Advisor: Professor Gregory L. Geoffroy

**Work Experience:**

October 2003 – present: (Lead Research Chemist) Technical team leader for hydrophobic / performance coatings development team. Responsibilities include technical development of product, intellectual property protection, and interactions with external partners.

October 1997 – September 2003: (Senior Principal Research Chemist / Lead Research Chemist) Technical team leader for low k materials development. Responsibilities included technical development of product, intellectual property protection, and interactions with external partners. Developed a fully integratable spin-on low k dielectric material that meets current industry demands for on wafer application. Responsible for CRADA with Sandia National Laboratory. Representative to Industrial Advisory Board at the Center for Micro-Engineered Ceramics at the University of New Mexico.

November 1994 – October 1997 (Principal Research Chemist) Increased leadership responsibility for project coordination, in addition to research role.

May 1990 – November 1994 (Senior Research Chemist) Responsible for preparation, ion exchange and evaluation of candidate materials for gas separation applications. Supervised technicians. Specifically, investigated the synthesis of aluminosilicate and non-aluminosilicate open framework materials, their ion exchange and structural properties. Developed relationships between compositional and structural influences on selective adsorption of molecules of interest for particular separations. Used that information to focus iterative preparations of materials with superior properties.

10/89-5/90 Research Assistant, UCSB

Investigated and developed novel organometallic routes to III-V semiconductors in crystalline and amorphous porous hosts. Studied solution loading techniques along with vapor phase loadings to improve homogeneity and stoichiometry in small III-V clusters. Investigated the effect of shape and charge of the host framework on the clusters formed. Used UV-Visible, Infrared, and Solid State Magic Angle Spinning (MAS) NMR spectroscopies, along with X-ray diffraction to characterize these novel inclusion compounds.

6/89-10/89 Summer Student, Central Research and Development Department, E. I. duPont deNemours & Co., Wilmington, DE; Supervisor: Dr. Norman Herron. Investigated organometallic routes to the synthesis of small particle III-V semiconductors, both in solution and in porous hosts. Attempted to extend known organometallic chemistry into porous glasses. Also, investigated novel routes to the synthesis of "Capped" III-V clusters in solution. Used UV-Visible and Solid State Magic Angle Spinning (MAS) NMR spectroscopies, along with X-ray diffraction to characterize these new materials.

1/88 - 6/89 Research and Teaching Assistant, UCSB.

Investigated and developed uses of organometallic precursors for synthesizing II-VI and III-V semiconductors encapsulated in crystalline and amorphous porous hosts. Characterized these materials using X-ray diffraction (both powder and single crystal), diffuse reflectance UV-Visible and Infrared spectroscopies, Solid State Magic Angle Spinning (MAS) NMR, Extended X-ray Absorption Fine Structure, and Degenerate Four-Wave-Mixing Spectroscopies. Directed the research of two undergraduate and two 1st year graduate students. Taught two quarters of undergraduate chemistry lab, and was teaching assistant one quarter for a senior level inorganic chemistry course.

7/87 - 1/88 Summer Student, Central Research and Development

Department, E. I. duPont deNemours & Co., Wilmington, DE; Supervisor: Dr. Norman Herron. Investigated routes to the synthesis of III-V semiconductors into porous hosts. Attempted to extend known organometallic chemistry into both glasses and zeolites for the synthesis of Size Quantized semiconductor clusters.

9/86- 6/87 Research and Teaching Assistant, UCSB.

Synthesized novel I-VII and II-VI clusters in sodalites. Characterized these materials by X-ray diffraction (both powder and single crystal), and Infrared (near and far) spectroscopy. Taught three quarters of undergraduate chemistry lab.

2/86 - 9/86 Summer Student, Central Research and Development

Department, E. I. duPont deNemours & Co., Wilmington, DE., Supervisor: Dr. Norman Herron. Investigated the synthesis and nonlinear optic properties of elemental and II-VI semiconductors in molecular sieves. Characterized Se and II-VI semiconductor clusters encapsulated in zeolites by powder X-ray diffraction, UV-Visible and Infrared, Solid State MAS-NMR, Second Harmonic Generation (SHG), and Extended X-ray Absorption Fine Structure (EXAFS) spectroscopies.

6/85-12/85 Undergraduate Research Assistant, Pennsylvania State

University. Studied routes to early/late heterobimetallic phosphidobridged dimers of Ta(V) and Fe, W, Pt, and Rh. Characterized these dimers by liquid state NMR, Infrared, UV-Visible, and Mass spectroscopies.